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(54) PRODUCTION OF HIGH-PURITY SILICON SINGLE CRYSTAL AND HIGH-PURITY SILICON SINGLE CRYSTAL

(57)Abstract:

PURPOSE: To obtain high-purity silicon single crystal having a trace amt. of oxygen induced defects in production of the silicon single crystal by a CZ method or MCZ method.

CONSTITUTION: The concn. of the metallic impurities in the surface layer of a graphite crucible 2 fitted to the outer side of a quartz crucible 3 is confined to ≤ 200 ppmwt and the concn. of the metallic impurities included in the entire part of the graphite crucible 2 is confined to ≤ 2 ppmwt. The pulling up of the silicon single crystal 8 is executed by managing the concn. of the metallic impurities of the graphite crucible 2 in such a manner, by which the amt. of the metallic elements evaporating from the graphite crucible 2 in a high-temp. state is drastically decreased and the clean silicon single crystal having the substantially negligible oxygen induced defects is produced.

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CLAIMS

[Claim(s)]

[Claim 1] The manufacture approach of the high-purity-silicon single crystal characterized by setting to 2 or less ppmwts metal high impurity concentration which sets to 200 or less ppmwts metal high impurity concentration in the surface of the carbon material structure used for the elevated-temperature part in a furnace in the silicon single crystal manufacturing installation by the Czochralski method which impresses the Czochralski method or a magnetic field, and is contained in said carbon material structure.

[Claim 2] The number of oxygen induction defects is five-piece cm² at the average. The following and maximum are 100-piece cm². n mold high-purity-silicon single crystal by the manufacture approach of the high-purity-silicon single crystal of claim 1 characterized by being the following.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to the high-purity-silicon single crystal manufactured using the manufacture approach of the high-purity-silicon single crystal by the Czochralski method which impresses the Czochralski method or a magnetic field, and said manufacture approach.

[0002]

[Description of the Prior Art] Although the high purity silicon is mainly used for the substrate of a semiconductor integrated circuit component, this high purity silicon is single-crystal-ized, the CZ process, i.e., MCZ method etc., for impressing the Czochralski method (henceforth a CZ process), and a magnetic field etc. In the single crystal which was able to be pulled up using the CZ process or the MCZ method, oxygen is dissolving and this oxygen has played the important role in the substrate manufacture process of a semiconductor integrated circuit component. That is, the oxygen in a silicon single crystal catches the metal impurity mixed in a component manufacture process (gettering), and raises the electrical property of a component. To the oxygen density, since it is very sensitive, said gettering operation occupies the important location on manufacture of the silicon single crystal according [control of an oxygen density] to a CZ process or the MCZ method.

[0003] In silicon melt, the oxygen into which the oxygen in a silicon single crystal deposited from the quartz crucible is incorporated by penetration at the time of raising of a silicon single crystal, and this is incorporated in a solid-state. The oxygen which melted into melt from the quartz crucible moves by the heat convection in the inside of melt. It is known that 90% or more of the oxygen which melted into said melt will evaporate as silicon monoxide (SiO) from a melt front face (refer to reference-1:LANDOLT-BORNSTEIN, EDS:K-H.HELLWEGE, and O.MADELING VOL.17 (1984) 40 SPRINGER-VERLAG).

[0004] The quality improvement demand of the silicon single crystal by the CZ process or the MCZ method is still severer with the improvement in a degree of integration of a semiconductor integrated circuit component. Since especially an oxygen induction defect (it is also called OSF) is a thing which makes a substrate surface layer produce a defect, it considers that the reduction influences the yield of an integrated circuit device directly. Since said oxygen induction defect was produced when the silicon wafer which carried out intentionally contamination was heat-treated, it became clear that it is what is produced when a metal exists in a silicon single crystal periodically [in recent years] (refer to reference-2:ELECTROCHEMICAL SOCIETY, 169TH SOCIETY MEETING EXTENDED ABSTRACT VOL.86-1, and 372 (1986)). Therefore, reduction of a metallic element has been an important technical problem on high quality silicon single crystal manufacture.

[0005] It was thought that two or more pollution sources in the silicon single crystal raising process by the CZ process existed conventionally. That is, it is a quartz crucible (an impurity melts into silicon melt during growth of a single crystal) as the 3rd pollution source of an impurity (3) which deals with it with crushing of polycrystalline silicon, and sometimes adheres as the 2nd pollution source of polycrystalline silicon (2) as the (1) 1st pollution source.

(4) Since the flow of inert gas prevents the back diffusion of electrons to melt even if there is evaporation of the impurity from a part which is a furnace atmosphere under silicon single crystal raising as the 4th pollution source, and was heated about the above (4) among these pollution sources by the elevated temperature in a furnace, it is supposed that it will not become a problem (said reference -1 reference). And research has been chiefly done about the pollution source of the above (3).

[0006]

[Problem(s) to be Solved by the Invention] Although carbon material is generally used for the hot zone

parts, for example, outside crucible, of a single crystal manufacturing installation by the CZ process, it is considered that the contamination by these carbon material is what can be disregarded from the former (said reference -1 reference). However, also although it is called the carbon material of a high grade, the impurity of ppm order is included. And the carbon material in a furnace of the single crystal manufacturing installation by (1) CZ process reacts with the silicon monoxide which evaporates briskly from silicon melt as shown below.

$2C(\text{solid-state}) + SiO(\text{gas}) \rightarrow CO(\text{gas}) + SiC(\text{solid-state})$

(2) If it contacts, the carbon material, i.e., the graphite crucible, which are a quartz crucible and its supporter under an elevated temperature, oxidation reaction of carbon material will be produced too and a carbon monoxide (CO) will be generated. Consequently, the metal metallurgy group oxide which gasifies a quartz and carbon material and was not able to be gasified is accumulated between a quartz crucible and a graphite crucible, and raises the concentration.

(3) In addition to the above, the metal impurity in the quartz crucible which is storing silicon melt moves into silicon melt with fusion of a quartz crucible, evaporates as a metallic oxide from a melt side, and adheres in a furnace. The metallic element adhering to the elevated-temperature section evaporates again, it is incorporated in a raising single crystal, and reducing the quality of a single crystal is predicted.

[0007] In order to confirm the above-mentioned consideration, the amount of the metal impurity contained in the surface of the graphite crucible used many times was measured. First, the surface of the graphite crucible used many times was shaved off, the acid extracted the metal contained in the surface, and this was analyzed using atomic absorption equipment and inductively-coupled-plasma luminescence equipment. In said surface, the amounts of metallic elements, such as Fe, Cr, and nickel, are increasing rapidly with use count increase of a graphite crucible. Next, in order to know the depth direction distribution of these metallic elements, it investigated using secondary-ion-mass-spectroscopy equipment about distribution of the metal impurity of the depth direction which goes to the interior from the front face of the graphite crucible before use and the graphite crucible after use. Consequently, it turned out that it is decreasing as metal high impurity concentration is high concentration near the front face of a graphite crucible and it goes to the interior. The range of the high concentration field of a metal impurity is several mm toward the interior from a front face. Moreover, when the consistency of the oxygen induction defect generated in a raising single crystal is measured, said consistency is increasing rapidly with the increment in the metal high impurity concentration of a graphite crucible.

[0008] Evaporation of the impurity from a part heated by the elevated temperature within the contents these experimental results are indicated to be by said reference -1, i.e., a furnace. It is a thing contrary to the conventional judgment it is supposed that it has not been a problem since the flow of inert gas can protect the back diffusion of electrons to melt. It is shown that it is a thing based on contamination by the metal impurity evaporation from the carbon material by which the oxygen induction defective generating cause in a raising single crystal is exposed to the elevated temperature, especially a graphite crucible etc. These are having discovered for the first time in high quality high-purity-silicon single crystal development of this invention person etc. and a research process. This invention was made based on the conventional trouble described previously, and this invention person's etc. consideration and experimental result, and aims at offering the manufacture approach of a high-purity-silicon single crystal and high-purity-silicon single crystal which controlled generating of an oxygen induction defect.

[0009]

[Means for Solving the Problem] In order to attain the above-mentioned purpose, the manufacture approach of the high-purity-silicon single crystal concerning this invention In the silicon single crystal manufacturing installation by the CZ process or the MCZ method, metal high impurity concentration in the surface of the carbon material structure used for the elevated-temperature part in a furnace is set to 200 or less ppmwts. And for the silicon single crystal which shall set to 2 or less ppmwts metal high impurity concentration contained in said carbon material structure, and is manufactured by such approach, the number of oxygen induction defects is five-piece cm^2 at the average. The following and maximum are 100-piece cm^2 . It considered as n mold high-purity-silicon single crystal which is the following.

[0010]

[Function] Since the metal high impurity concentration of the carbon material structure used for the elevated-temperature part in a furnace was restricted to 2 or less ppmwts by the whole which contains 200 or less ppmwts and a surface in a surface according to the above-mentioned configuration, when a silicon single crystal is raised using such carbon material, generating of an oxygen induction defect can fully be controlled. And it sets to n mold high-purity-silicon single crystal by the manufacture approach of the high-

purity-silicon single crystal by this invention, and is five-piece cm² at the average about the number of oxygen induction defects. It is 100-piece cm² at the following and maximum. It can stop below.

[0011]

[Example] Below, the manufacture approach of the high-purity-silicon single crystal concerning this invention and the example of n mold high-purity-silicon single crystal by this manufacture approach are explained with reference to a drawing. The quartz crucible 3 is attached in the inside of a graphite crucible 2 established in the chamber 1, drawing 1 is drawing showing the construct of the silicon single crystal manufacturing installation by the CZ process, the graphite heater 4 is formed so that said graphite crucible 2 may be surrounded, and the heat insulating mould 5 is installed in the periphery. The thing of 2 or less ppmwts is used for the metal high impurity concentration of the whole in which the metal high impurity concentration in a surface is 200 or less ppmwts, and, as for said graphite crucible 2, contains a surface. That is, the metal high impurity concentration of the graphite crucible 2 at the time of a new article is stopped by 2 or less ppmwts regardless of a surface and the whole, and although the metal impurity which deposits near a surface with the increment in a use count increases, as for the metal high impurity concentration in a surface, only the thing of 200 or less ppmwts will always be used. In addition, as for a crucible shaft and 7, 6 is [melt and 8] the silicon single crystals under training.

[0012] While carrying out heating fusion of the raw material with which the quartz crucible 3 was filled up at the graphite heater 4, phosphorus was added as dope material. The seed crystal attached in the seed shaft being immersed in said melt 7, and rotating a seed shaft and a graphite crucible 2 to this direction or hard flow, the seed shaft was pulled up and the silicon single crystal 8 was grown up. Thus, the body section of the obtained silicon single crystal was cut and divided, it heat-treated by 1100-degreeC within the dry oxygen ambient atmosphere for 16 hours, after immersion and rinsing and a microscope were used for the JIRUTORU-etching reagent, and the number of oxygen induction defects of each cutting plane was measured. When the sum total of the number of oxygen induction defects within the 250 time microscopic field was made into maximum and the average value of the number of oxygen induction defects within said microscopic field measured about five on a single crystal diameter, respectively was made into the average, at maximum, the number of oxygen induction defects of said single crystal was 100 or less pieces, and the average value was five or less pieces. That is, to the number of oxygen induction defects by the conventional manufacture approach having been thousands, it means decreasing sharply to some and the oxide-film pressure resistance of a silicon single crystal improves remarkably.

[0013]

[Effect of the Invention] As explained above, since [according to this invention] the metal high impurity concentration of the carbon material structure used for the elevated-temperature part in a furnace, especially a graphite crucible is restricted to 2 or less ppmwts by the whole which contains 200 or less ppmwts and a surface in a surface in the silicon single crystal manufacturing installation by the CZ process or the MCZ method and a silicon single crystal is raised, generating of the oxygen induction defect in a raising single crystal can fully be controlled. Consequently, n mold high-purity-silicon single crystal which held down the number of oxygen induction defects to five or less pieces, and held down maximum to 100 or less pieces by the average can be manufactured.

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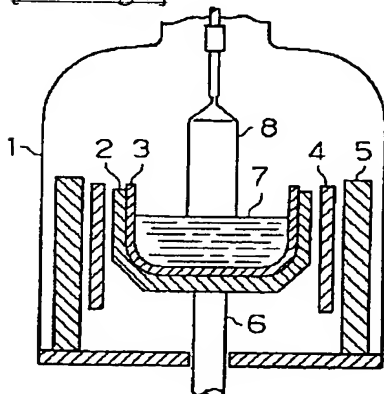
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DRAWINGS

[Drawing 1]



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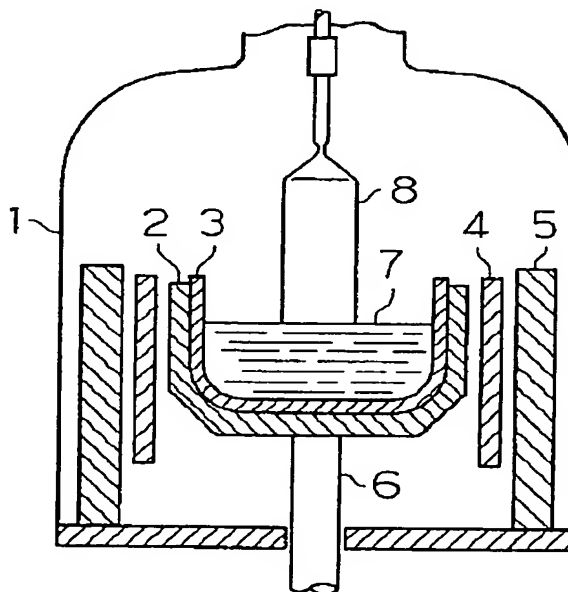
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(54)【発明の名称】 高純度シリコン単結晶の製造方法および高純度シリコン単結晶

(57)【要約】

【目的】 CZ法またはMCZ法によるシリコン単結晶の製造において、酸素誘起欠陥の微量な、高純度シリコン単結晶を得ることができるようにする。

【構成】 石英るつぼ3の外側に嵌着する黒鉛るつぼ2の表層における金属不純物濃度を200ppmw以下とし、かつ、前記黒鉛るつぼ2全体に含まれる金属不純物濃度を2ppmw以下とする。このように黒鉛るつぼ2の金属不純物濃度を管理してシリコン単結晶8の引き上げを行うことにより、高温状態で黒鉛るつぼ2から蒸発する金属元素量が激減し、実質的に酸素誘起欠陥を無視し得る清浄なシリコン単結晶を製造することができる。



【特許請求の範囲】

【請求項1】 チョクラスキー法または磁場を印加するチョコラルスキー法によるシリコン単結晶製造装置において、炉内高温部分に使用する炭素材構造物の表層における金属不純物濃度を200ppmw以下とし、かつ、前記炭素材構造物に含まれる金属不純物濃度を2ppmw以下とすることを特徴とする高純度シリコン単結晶の製造方法。

【請求項2】 酸素誘起欠陥数が平均値で5個 cm^2 以下、かつ、最大値が100個 cm^2 以下であることを特徴とする請求項1の高純度シリコン単結晶の製造方法によるn型高純度シリコン単結晶。

【発明の詳細な説明】

【0001】

【産業上の利用分野】本発明は、チョコラルスキー法または磁場を印加するチョコラルスキー法による高純度シリコン単結晶の製造方法および前記製造方法を用いて製造する高純度シリコン単結晶に関する。

【0002】

【従来の技術】半導体集積回路素子の基板には、主として高純度シリコンが用いられているが、この高純度シリコンは、チョコラルスキー法（以下CZ法という）や磁場を印加するCZ法すなわちMCZ法などによって単結晶化される。CZ法またはMCZ法を用いて引き上げられた単結晶中には酸素が固溶しており、この酸素が半導体集積回路素子の基板製造プロセスにおいて重要な役割を果たしている。すなわち、シリコン単結晶中の酸素は、素子製造プロセスで混入する金属不純物を捕捉（ゲッターリング）し、素子の電気特性を向上させる。前記ゲッターリング作用は、酸素濃度に対して極めて敏感であるため、酸素濃度の制御はCZ法またはMCZ法によるシリコン単結晶の製造上重要な位置を占めている。

【0003】シリコン単結晶中の酸素は、石英るつぽから析出した酸素がシリコン融液中に溶け込み、これがシリコン単結晶の引き上げ時に固体中に取り込まれたものである。石英るつぽから融液中に溶け込んだ酸素は、熱対流により融液内を移動する。前記融液中に溶け込んだ酸素の90%以上は、融液表面から一酸化珪素（SiO）として蒸発することが知られている（文献-1：L ANDOLT-BORNSTEIN, EDS：K-H. HELLWEGE, O. MADELING VOL. 17（1984）40 SPRINGER-VERLAG 参照）。

【0004】CZ法またはMCZ法によるシリコン単結晶の高品質化要求は、半導体集積回路素子の集積度向上に伴ってますます厳しいものとなっている。特に、酸素誘起欠陥（OSFともいう）は基板表面層に欠陥を生じさせるものであるため、その低減は集積回路素子の歩留りに直接影響すると見なされている。前記酸素誘起欠陥は、故意汚染したシリコンウェーハを加熱処理すると生

じることから、金属がシリコン単結晶中に存在することによって生じるものであることが近年定期的に明らかになった（文献-2：ELECTROCHEMICAL SOCIETY, 169TH SOCIETY MEETING EXTENDED ABSTRACT VOL. 86-1,（1986）372 参照）。従って、金属元素の低減が高品質シリコン単結晶製造上重要な課題となっている。

【0005】従来、CZ法によるシリコン単結晶引き上げ過程での汚染源は複数個存在すると考えられていた。すなわち、

（1）第1の汚染源として、多結晶シリコン

（2）第2の汚染源として、多結晶シリコンの破碎と取扱いに付着する不純物

（3）第3の汚染源として、石英るつぽ（単結晶の成長中にシリコン融液中に不純物が溶け込む）

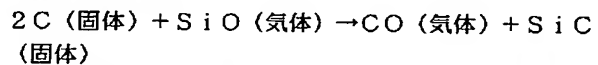
（4）第4の汚染源として、シリコン単結晶引き上げ中における炉内雰囲気

であり、これらの汚染源のうち上記（4）については、炉内の高温に加熱された部分からの不純物の蒸発があっても、不活性ガスの流れが融液への逆拡散を防ぐので、問題にはならないとされている（前記文献-1参照）。そして、もっぱら上記（3）の汚染源について研究が行われてきた。

【0006】

【発明が解決しようとする課題】CZ法による単結晶製造装置のホットゾーンパーツたとえば外側るつぽには、一般に炭素材が使用されているが、これらの炭素材による汚染は従来から無視しうるものと見なされてきた（前記文献-1参照）。しかしながら、高純度の炭素材といえどもppmオーダーの不純物を含んでいる。そして、

（1）CZ法による単結晶製造装置の炉内炭素材は、下記に示すようにシリコン融液から盛んに蒸発する一酸化珪素と反応する。



（2）高温下で石英るつぽとその保持体である炭素材すなわち黒鉛るつぽが接触すると、やはり炭素材の酸化反応を生じ、一酸化炭素（CO）が発生する。その結果、石英および炭素材がガス化し、ガス化しきれなかった金属や金属酸化物が石英るつぽと黒鉛るつぽとの間に蓄積し、その濃度を高める。

（3）上記に加えて、シリコン融液を貯留している石英るつぽ中の金属不純物は、石英るつぽの融解とともにシリコン融液中に移動し、融液面から金属酸化物として蒸発し、炉内に付着する。高温部に付着した金属元素は再び蒸発し、引き上げ単結晶中に取り込まれ、単結晶の品質を低下させることが予測される。

【0007】上記考察を確かめるため、多数回使用した黒鉛るつぽの表層に含まれる金属不純物の量を測定し

た。まず、多数回使用した黒鉛るつぼの表層を削り取り、表層に含まれている金属を酸により抽出し、これを原子吸光装置および誘導結合プラズマ発光装置を用いて分析した。前記表層においては、黒鉛るつぼの使用回数増大に伴ってFe、Cr、Ni等の金属元素量が急増している。次に、これらの金属元素の深さ方向分布を知るために、使用前の黒鉛るつぼと使用後の黒鉛るつぼの表面から内部に向かう深さ方向の金属不純物の分布について、二次イオン質量分析装置を用いて調査した。その結果、金属不純物濃度は黒鉛るつぼの表面近傍で高濃度になっており、内部に向かうに従って減少していることが分かった。金属不純物の高濃度領域は、表面から内部に向かって数mmの範囲である。また、引き上げ単結晶に発生する酸素誘起欠陥の密度を測定したところ、前記密度は黒鉛るつぼの金属不純物濃度の増加とともに急増している。

【0008】これらの実験結果は、前記文献-1に記載されている内容、すなわち炉内の高温に加熱された部分からの不純物の蒸発は、不活性ガスの流れにより融液への逆拡散を防ぐことができるので問題になっていないとする従来の見解に反するものであり、引き上げ単結晶中の酸素誘起欠陥発生原因が高温に晒されている炭素材、特に黒鉛るつぼからの金属不純物蒸発等による汚染に基づくものであることを示している。これらは、本発明者等の高品質高純度シリコン単結晶開発、研究過程で初めて発見したことである。本発明はさきに述べた従来の問題点と、本発明者等の考察ならびに実験結果に基づいてなされたもので、酸素誘起欠陥の発生を抑制した高純度シリコン単結晶の製造方法および高純度シリコン単結晶を提供することを目的としている。

【0009】

【課題を解決するための手段】上記目的を達成するため、本発明に係る高純度シリコン単結晶の製造方法は、CZ法またはMCZ法によるシリコン単結晶製造装置において、炉内高温部分に使用する炭素材構造物の表層における金属不純物濃度を200ppmw以下とし、かつ、前記炭素材構造物に含まれる金属不純物濃度を2ppmw以下とするものとし、このような方法で製造するシリコン単結晶は、酸素誘起欠陥数が平均値で5個/cm²以下、かつ、最大値が100個/cm²以下であるn型高純度シリコン単結晶とした。

【0010】

【作用】上記構成によれば、炉内高温部分に使用する炭素材構造物の金属不純物濃度を、表層において200ppmw以下、表層を含む全体では2ppmw以下に制限したので、このような炭素材を用いてシリコン単結晶の引き上げを行った場合、酸素誘起欠陥の発生を十分に抑制することができる。そして、本発明による高純度シリコン単結晶の製造方法によるn型高純度シリコン単結晶においては、酸素誘起欠陥数を平均値で5個/cm²以

下、かつ、最大値で100個/cm²以下に抑えることができる。

【0011】

【実施例】以下に、本発明に係る高純度シリコン単結晶の製造方法およびこの製造方法によるn型高純度シリコン単結晶の実施例について、図面を参照して説明する。図1はCZ法によるシリコン単結晶製造装置の構成概念を示す図で、チャンバ1内に設けた黒鉛るつぼ2の内面に石英るつぼ3が嵌着され、前記黒鉛るつぼ2を包囲するように黒鉛ヒータ4が設けられ、その外周に保温筒5が設置されている。前記黒鉛るつぼ2は、表層における金属不純物濃度が200ppmw以下で、かつ、表層を含む全体の金属不純物濃度が2ppmw以下のものが使用される。すなわち、新品時の黒鉛るつぼ2の金属不純物濃度は表層、全体を問わず2ppmw以下に抑えられており、使用回数の増加に伴って表層付近に析出する金属不純物が増加するが、表層における金属不純物濃度は常に200ppmw以下のものののみが使用されることになる。なお、6はるつぼ軸、7は融液、8は育成中のシリコン単結晶である。

【0012】石英るつぼ3に充填した原料を黒鉛ヒータ4によって加熱溶融するとともにドーパ材として燐を添加した。シード軸に取り付けた種子結晶を前記融液7に浸漬し、シード軸および黒鉛るつぼ2を同方向または逆方向に回転しつつシード軸を引き上げてシリコン単結晶8を成長させた。このようにして得られたシリコン単結晶の直胴部を切断、分割し、ドライ酸素雰囲気内で1100°Cで16時間熱処理し、ジルトルーエッチング液に浸漬、水洗の後、顕微鏡を用いて各切断面の酸素誘起欠陥数を測定した。250倍顕微鏡視野内の酸素誘起欠陥数の合計を最大値とし、単結晶直径上の5点についてそれぞれ測定した前記顕微鏡視野内の酸素誘起欠陥数の平均値をアベレージとすると、前記単結晶の酸素誘起欠陥数は最大値で100個以下、平均値は5個以下であった。すなわち、従来の製造方法による酸素誘起欠陥数が数千であったのに対して、数個に激減したことになり、シリコン単結晶の酸化膜耐圧性が著しく向上する。

【0013】

【発明の効果】以上説明したように本発明によれば、CZ法またはMCZ法によるシリコン単結晶製造装置において、炉内高温部分に使用する炭素材構造物、特に黒鉛るつぼの金属不純物濃度を、表層において200ppmw以下、表層を含む全体では2ppmw以下に制限してシリコン単結晶の引き上げを行うこととしたので、引き上げ単結晶における酸素誘起欠陥の発生を十分に抑制することができる。その結果、酸素誘起欠陥数を平均値で5個以下、最大値を100個以下に抑えたn型高純度シリコン単結晶を製造することができる。

【図面の簡単な説明】

【図1】CZ法によるシリコン単結晶製造装置の構成概

(4)

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念を示す図である。

【符号の説明】

2 黒鉛るつば

* 3 石英るつば

7 融液

* 8 シリコン単結晶

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【図1】

